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(54) Title: PROCESS FOR PREPARING MICROCAPSULES CONTAINING A FLAVOURANT EMBEDDED IN A MATRIX MATERIAL AND PRODUCTS PREPARED BY USING THE PROCESS

(57) Abstract

Process for preparing microcapsules comprising a flavourant embedded in a matrix material comprising spraying an aqueous emulsion, suspension or solution of the flavourant and the matrix material under the supply of air having a temperature of 50-120 °C while simultaneously introducing a spraying agent, and subsequently treating the sprayed product in a fluidized bed at a temperature of 0-120 °C to form a free-flowing product.

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# PROCESS FOR PREPARING MICROCAPSULES CONTAINING A FLAVOURANT EMBEDDED IN A MATRIX MATERIAL AND PRODUCTS PREPARED BY USING THE PROCESS

The present invention relates to a process for preparing microcapsules containing a flavourant embedded in a matrix material comprising spraying an aqueous emulsion, suspension or solution of the flavourant and the matrix material under the supply of air to form a free-flowing product.

Microcapsules containing a flavourant having fragrance and/or taste properties are used in a variety of products, such as foods and drinks, confectionery products, medical products, tooth paste, detergents, cosmetic and sanitary articles.

Irrespective of the area of use it is important that the microcapsules are stable by which is meant that the content of flavourant is maintained substantially unchanged. This implies that the flavourant must not escape or be converted, e.g. by reaction with the oxygen of the air or by reaction with other substances.

Herein flavourants mean substances having fragrance and/or taste properties. Flavourants are typically volatile compounds which tend to evaporate quickly unless they are encapsulated effectively in a non-reactive encapsulant.

Various processes for preparing microcapsules of the type mentioned above for use in chewing gum are known. When using such microcapsules in chewing gum the aim is to obtain a gradual release of the flavourant and control of the release so that the chewing gum maintains its flavour even when it is chewed for longer periods of time, such as several minutes.

US patent specification No. 4,386,106 discloses a process wherein the matrix material comprises gelatine, natural gum and a plasticizer and wherein an aqueous emulsion of a flavourant and the matrix material is roll dried to form a solid mass which is subsequently ground to form a powder, and the powder formed is coated with a water-insoluble material consisting of polyvinyl acetate, polyvinyl alcohol, zein or ethyl cellulose.

WO 89/05590 discloses a method of making a chewing gum containing wax embedded ingredients, including powdered flavourants to obtain a delayed release of the wax embedded ingredients which are incorporated in the chewing gum in comminuted particle form.

It is also well known to prepare a free-flowing powder containing a flavourant by spray drying and by using a matrix material consisting of gelatine or natural gum and a carbohydrate, such as saccharose, fructose and corn sirup.

However, a powder containing a flavourant and prepared by a conventional spray drying method does not provide sufficient protection of the flavourant to prevent part of the flavourant from escaping during the preparation and before use of the powder, or as composite flavourants are often used, such as e.g. orange oil, the more volatile component will escape and the composite flavourant will change character as a result thereof. The particles formed are porous and do not have a sufficiently high resistance against oxygen to prevent the oxygen of the air from reacting with the flavourant and converting parts of said flavourant into undesired compounds, see i.a. Taylor, A. H. (1983), Encapsulation systems and their applications in the flavour industry. Food Flav. Ingr. Proc. Pckg, Sep. 1983, pp. 48-52.

Surprisingly, it has now been found that the drawbacks of the prior art spray drying methods can be reduced or eliminated by the process according to the invention, which process is characterized in that the aqueous emulsion, suspension or solution of a flavourant and matrix material is sprayed under the supply of air having a temperature of 50 to 120°C while simultaneously introducing a spraying agent, and in that the sprayed product is subjected to an additional drying in a fluidized bed at a temperature between 0 and 120°C.

In conventional spray drying hot air having a temperature of 180-300° is typically supplied. When the liquid particles formed by the spraying meet the very hot air, a steam explosion occurs which results in that the matrix material in which the flavourant is enclosed becomes porous and permeable to flavourant and oxygen. The

product formed typically has a particle density of from 0.7 to 0.9 g/ml.

In the process of the invention comprising drying the liquid drops at a much lower temperature a considerably more dense matrix material is obtained, said matrix material having a particle density of from 1.0 to 1.5 g/ml, preferably from 1.1 to 1.4 g/ml, and as a result thereof it becomes substantially non-permeable to both the flavourant enclosed in the matrix material and to the oxygen contained in the ambient air.

A further advantage of the process according to the invention is that the solid particles formed by the spray drying are spherical as opposed to the particles formed by conventional spray drying which are irregular. As a result of the regular spherical shape the surface area is smaller which results in a reduced exudation of the flavourant from the microcapsules. Furthermore a smaller part of the flavourant will be deposited on the surface of the particles than by conventional spray drying.

Furthermore, it is possible to obtain larger particles with the process according to the invention and hence a reduced relationship between the surface area and the volume compared to the conventional spray drying, said reduced relationship contributing to reduce the evaporation. Thus, the process according to the invention allows the preparation of microcapsules having a size interval in the range of from 50-800 μm, whereas the particle size interval for particles prepared by the prior art processes ordinarily is considerably more narrow and typically varies in the range of from 10 to 100 μm.

Moreover, it has surprisingly been found that by using an emulsion which furthermore comprises wax it is possible to obtain a microencapsulated product containing a flavourant, said product exhibiting a considerably extended flavour release in confectionery products, such as chewing gum.

The flavourants used in the process according to the invention are water in-soluble in most uses and may contain functional groups, such as alcohols, ethers, aldehydes, acetales, ketones, esters and

lactones and comprise e.g. terpenes, heterocyclic compounds and mixtures thereof having molecular weights of from approx. 100 to approx. 300 and having a boiling point below 180°C, or the flavourants may be constituted by essential oils having a content of the low molecular compounds mentioned above, such as anise oil, pine needle oil, jasmin oil, camphorated oil, wintergreen oil, lavender oil, melissa oil, rosemary oil, eucalyptus oil, citrus oils and peppermint oil and spice oils and extracts of spice oils, such as extracts of parsley oil, lovage oil and dill oil.

Examples of water-soluble flavourants are acidulants, such as citric acid, tartaric acid, malic acid, orthophosphoric acid; sweeteners, such as glycyrrhizin and glycyrrhizinate, and bitter substances, such as coffein.

As matrix material in the process according to the invention colloids may be used, such as vegetable gum, e.g. gum arabic, and tragacanth gum, starch, including modified starch, dextrins, proteins, e.g. gelatine, including gelatine recovered from fish and other marine animals, succinylated gelatine, soya protein and caseinates. Furthermore the matrix material may include saccharides, such as saccharose, sugar alcohols and synthetic sweeteners and mixtures thereof. An example of a preferred matrix former is gelatine having a Bloom strength of from 0 to 300. The selection of matrix material depends on the flavourant used. For instance different types of gum arabic produce different permeability.

In order to obtain a slow release of flavourant from the microcapsules the matrix material according to a preferred embodiment may further contain wax, preferably relatively hard, water-insoluble wax having a low penetrometer resistance and a melting point which is lower than the boiling point of the flavourant. In an emulsion or suspension the following proportion is used : from 1-15% by weight of flavourant, from 20-35% by weight of colloid and from 1-8% by weight of wax, and the wax constitutes from 5 to 20% by weight of the finished microcapsules, preferably about 10% by weight.

The waxes used may be natural waxes (wax esters), such as carnauba

wax and candelila wax or synthetic waxes which are typically long-chained carbonhydrides, such as petroleum wax, paraffin wax, Hartwachs SP1044 supplied by Dansk Voksfabrik, synthetic waxes, polyethylene wax, polyethylene glycol and polyethylene oxide having a high molecular weight, etc. or stearin. It is preferred to use hard, microcrystalline waxes having a melting point of from approx. 40° to approx. 120°C, preferably from about 60° to 90°C.

Additives, such as emulsifiers, complexing agents, plasticizers and antioxidants may be used in the matrix materials mentioned above.

As spraying agent in the process according to the invention hydrophilic agents which furthermore act as contact drying agents may be used and which may constitute a relatively large part of the product (up to 30%), such as starch, modified starch, tricalcium phosphate, lactose, mannitol, ethyl cellulose, coagulated albumin and cured gelatine, or hydrophobic agents which constitute a relatively small part of the product (2-10%) may be used , such as casein, stearat-Ca and stearat-Na, hydrogenated castor oil, polyoxide, talc, vegetable wax, preferably carnauba wax and candelila wax, and paraffin wax, preferably a wax marketed under the name Hartwachs SP1044. The waxes are preferably microcrystalline. A minimum of 1 part by weight of spraying agent is used per part by weight emulsion, suspension or solution. The melting point of the spraying agent is preferably higher than 40°C, most preferable from 80°C-130°C.

In the preparation of a microencapsulated flavour product for use in confectionery products, such as chewing gum, where a flavour extension is desired it is preferred to use a spraying agent which is relatively hydrophobic and/or insoluble in water, such as casein, wax, paraffin (petroleum wax). As a result thereof the solvent effect of the saliva on the microcapsules is reduced.

Theoretically an emulsion, suspension or solution containing volatile flavourants will lose constituents which are highly volatile in connection with a spraying in a hydrophobic spraying agent. On the other hand more compact particles having a higher total content of flavourant can be obtained after drying is

completed.

In a preferred embodiment of the process according to the invention an emulsion of an oil containing a flavourant is sprayed under oxygen-free conditions in an aqueous colloid solution which optionally may contain water-soluble flavourants, the oils drops having a diameter of from 0.2 to 10 μm, preferably about 1 μm. By using an emulsion of an oil containing a flavourant as starting material it is possible to prepare products having a content of flavourant of from 0.1-40% by weight, preferably from 15-20% by weight. A proportion of from 1/99 to 2/3 parts by weight of flavourant per part by weight of matrix material is preferably used. In a particularly preferred process according to the invention an emulsion which furthermore comprises wax is used.

The emulsion is preferably heated to between 20 and 80°C, adjusted to a viscosity of from 80 - 250 cP and sprayed into fine drops (diameter about 0.1 - 1.2 mm) by means of e.g. a disc sprayer in a spraying tower where the emulsion, at the moment of spraying, meets air having a temperature of between 50 and 120°C. As a result thereof the outer part of the matrix material in the drops solidifies, thereby forming a shell so that the particles no longer are permeable to the flavourants but so that water is still allowed to diffuse. The formation of the shell may optionally be expedited further by blowing a spraying agent in the form of e.g. corn starch into the spraying tower. In case it is not desired that the spraying agent constitutes a greater amount in the shell formation, a spraying agent such as calcium stearate, a wax marketed under the name Hartwachs SP1044 or hydrogenated caster oil can be used.

In a further preferred embodiment of the process according to the invention an emulsion of an oil containing a flavourant and an aqueous colloid solution is sprayed under the supply of air having an temperature of 60 - 85°C and the sprayed product is subsequently subjected to a further treatment in a fluidized bed at a temperature of between 20 and 85°C.

The drying of the product in a fluidized bed is preferably effected with dehumidified air. The drying time is between 1 and 10 hours

depending on the bach size to obtain a product having a water content of from aprrox. 2 to approx. 7%.

Furthermore the invention relates to microcapsules comprising a flavourant embedded in a matrix material, the microcapsules being prepared by spraying an aqueous emulsion, suspension or solution of flavourants and the matrix material under the supply of air having a temperature of 50 to 120°C while simultaneously introducing a spraying agent, and the sprayed product is subsequently subjected to a further drying in a fluidized bed at a temperature of between 0 and 120°C.

Finally the invention relates to a chewing gum containing the microcapsules mentioned above.

Example 1

Microencapsulation of orange oil.

2000 g of Water was heated to 60°C, 2000 g of modified starch ("N-Lok®") was added and dissolved under stirring.

The solution was deaerated under vacuum at 0.8 bar at 65°C for 2 hours. Vacuum in the container was reduced by introducing nitrogen and subsequently the solution was cooled to 13°C, and 1000 g of orange oil was added slowly under vigoruos stirring. The orange oil was added by pressing out the orange oil from the raw material container using nitrogen at 0.1-0.5 bar.

The orange oil was emulsified in the N-Lok® solution within 1 hour. During the oil emulsification the viscosity was maintained at about 450 cP.

The emulsion container was heated to 40°C and the viscosity was adjusted to 112 cP by the addition of water which was deaerated for oxygen.

The emulsion was introduced into a spraying tower at a velocity of 2 l/min. and sprayed therein in the form of fine drops (about 0.1-0.3

mm) in a cloud of fine particles of spraying agent in the form of corn starch which was supplied simultaneously to the spraying tower from a hopper at a velocity of 4 kg/min. The spraying was effected by means of a disc sprayer having 2500 rotations/min.

The temperature in the spraying tower was 66°C and during the passage down through the tower the surface of the emulsion drops solidified. The retention time in the spraying tower was estimated to 1 - 2 sec.

The emulsion drops having freshly solidified surfaces were transferred to a fluidized bed and maintained fluidized for about 2.5 hours by the introduction of a drying air having a temperature of 65°C. During the drying the excess corn starch was recycled to the hopper.

The ready-dried product was subsequently removed and sieved into two fractions, viz. a product fraction of 53-300μm and a coarse fraction. The product fraction constituted 98% of the dried product.

The product obtained was a free-flowing product having a high particle density and a content of orange oil of 17.1%. The product is soluble in cold water and suitable as a flavourant in soft drinks.

Example 2

Microencapsulation of butter flavour

700 g of Water was heated to 60°C, 438 g of dextrine ("Moresweet 1924®") and 292 g of gum arabic were added and dissolved under stirring. In order to provide the solution with a butter-like look 24 g of a 1% solution of betacarotene in water was added. In another container 15 g of Viscoleo® extra (coconut oil containing $C_{8-10}$ fatty acids) was mixed with 15 g of a liquid butter flavour, lot 15.94.251 supplied from International Flavors og Fragrances I.F.F. (Denmark) APS. In order to prevent oxidation a mixture of α-, β- and γ-tocopherol in an amount of 1% was added to the oil mixture.

The oil mixture was emulsified into the hot gum arabic/dextrin solution under nitrogen. The comminution of the oil drops was stopped at a size of 0.5-1 μm and the viscosity was adjusted to 120 cP at 60°C by dilution with water.

The emulsion was introduced into a spraying tower at a velocity of 1 l/min. and sprayed therein in the form of fine drops (about 0.3-0.8 mm) in a cloud of fine particles of the spraying agent which was supplied simultaneously to the tower from a hopper. The spraying agent was introduced at a velocity of 1.5 kg/min. The spraying was effected by means of a disc sprayer having 1200 rotations/min.

The temperature was 65°C in the spraying tower and during the passage down through the tower the surface of the fine drops solidified.

The emulsion drops having freshly solidified surfaces were transferred to a fluidized bed and maintained fluidized for about 1 hour by the introduction of drying air having a temperature of 65°C. During the drying the excess spraying agent was recycled to the hopper containing auxiliary agents.

The ready-dried product was removed and sieved into two fractions, viz. a product fraction of 250-820 μm and a coarse fraction of more than 820 μm.

The product obtained was a free-flowing product having a high particle density and a content of butter flavour of 1.5%. The product was dissolved in cold water at 10°C in less than 30 sec.

Example 3

Microencapsulation of eucalyptus oil

10 kg of Water was heated to 52°C, 4.446 kg of 240 Bloom type A gelatine and 2.223 kg of saccharose were added and dissolved in the water under stirring.

4.000 kg of eucalyptus oil having a content of 83% 1,8-cineol was

slowly emulsified into the gelatine/sugar solution under vigorous stirring. The comminution of the oil drops continued until the oil drops had a size of 1-2 μm.

The emulsion was subsequently heated to 70°C as the container was provided with a reflux condenser in order to avoid unneccesary loss of the eucalyptus oil at the high temperature. The viscosity was then adjusted to 180 cP by dilution with water.

The emulsion was now introduced into the spraying tower at a velocity of 2 l/min. and sprayed therein in the form of fine drops (about 0.2-0.3 mm) in a cloud of fine particles of spraying agent. The spraying agent was supplied to the spraying tower with a hot air stream of 85°C and in an amount of 3 kg/min. In order to ensure a rapid evaporation of water and to expedite the drying of the surface of the fine drops formed by spraying, additional air having a temperature of 85°C was supplied to the spraying tower in the spraying zone.

The spraying of the emulsion was effected by means of a disc sprayer having 2000 rotations/min. The temperature in the spraying tower was 65°C.

The emulsion drops having freshly solidified surfaces were transferred together with the spraying agent to a fluidized bed and maintained fluidized for about 2.5 hours by the introduction of drying air having a temperature of 63°C. During the drying the excess spraying agent was recycled to a hopper.

The ready-dried product was then removed and sieved into two fractions, viz. a product fraction of 53-300 μm and a coarse fraction. The product fraction constituted 97% of the dried product.

The product obtained was a free-flowing product having a high particle density and a content of eucalyptus oil of 17.7% and a content of 1,8-cineol of 14.62%. In other words a very insignificant loss of the most volatile component 1,8-cineol.

Example 4

Microencapsulation of mint oil

200 g of Water was heated to 60°C, 790 g of 240 Bloom type A gelatine was added and dissolved in water under stirring.

133.8 g of Carnauba wax was dissolved in 343 g of mint oil at 67-68°C to a homogeneous mixture.

The mint oil/wax mixture was slowly transferred to the gelatine/water solution under vigorous stirring. The comminution of the oil drops was continued until the oil drops had a size of 1 µm.

The viscosity was then adjusted to 180 cP by dilution with water.

The emulsion was now introduced into the spraying tower at a velocity of 3 l/min. and sprayed therein in the form of fine drops in a cloud of fine casein particles as spraying agent.

The spraying agent was supplied to the spraying tower with a hot air stream of 85°C and in an amount of 3-4 kg/min.

In order to ensure a rapid evaporation of water and to expedite the drying of the surface of the fine drops formed by spraying, additional air having a temperature of 85°C was supplied to the spraying tower in the spraying zone.

The spraying of the emulsion was effected by means of a disc sprayer having 2300 rotations/min. The temperature in the spraying tower was 65°C.

The emulsion drops having freshly solidified surfaces were transferred together with the spraying agent to a fluidized bed and maintained fluidized for about 3 hours by the introduction of drying air having a temperature of 63°C. During the drying the temperature of the encapsulated product did not exceed 40°C and the excess fine casein particles were recycled to a hopper.

The ready-dried product was then removed and sieved into two fractions, viz. a product fraction of 125-450 µm and a coarse fraction.

The product obtained was a free-flowing product having a high particle density of 1.2 g/ml.

The product is suitable for use in confectionery which requires an extension of flavour, e.g. chewing gum.

Example 5

Microencapsulation of spearmint oil

2000 g of Water was heated to 60°C, 790 g of 240 Bloom type A gelatine was added and dissolved in the water under stirring.

134 g of Carnauba wax was dissolved in 343 g of spearmint oil at 67°C.

The spearmint oil/wax mixture was slowly transferred to the gelatine/water solution under vigorous stirring. The comminution of the oil drops was continued until the oil drops had a size of 1 µm.

The viscosity was now adjusted to 160 cP by dilution with water.

The emulsion was then introduced into the spraying tower at a velocity of 3 l/min. and sprayed therein in the form of fine drops in a cloud of finely ground casein particles as spraying agent.

The spraying agent was supplied to the spraying tower with a hot air stream of 85°C and in an amount of 3-4 kg/min.

In order to ensure a rapid evaporation of water and to expedite the drying of the surface of the fine drops formed by spraying, additional air having a temperature of 85°C was supplied to the spraying tower in the spraying zone.

The spraying of the emulsion was effected by means of a disc sprayer

having 2300 rotations/min. The temperature in the spraying tower was 60°C.

The emulsion drops having freshly solidified surfaces were transferred together with the spraying agent to a fluidized bed and maintained fluidized for about 3 hours by the introduction of drying air having a temperature of 63°C. During the drying the temperature of the encapsulated product did not exceed 40°C and the excess finely ground casein particles were recycled to a hopper.

The ready-dried product was then removed and sieved into two fractions, viz. a product fraction of 125-450 µm and a coarse fraction.

The product obtained was a free-flowing product having a high particle density.

Example 6

Microencapsulation of orange oil

2000 g of Water was heated to 60°C, 790 g of 240 Bloom type A gelatine was added and dissolved in the water under stirring.

735 g of Lunacerin® W70 (paraffin wax) was melted at 73°C and 200 g of cold orange oil was added and the mixture was heated to 60°C.

The orange oil/wax mixture was slowly transferred to the gelatine/water solution under vigorous stirring. The comminution of the oil drops was continued until the oil drops had a size of 1 µm.

The viscosity was then adjusted to 160 cP by dilution with water.

The emulsion was then introduced into a spraying tower at a velocity of 3 l/min. and sprayed therein in the form of fine drops in a cloud of fine wax particles (Hartwachs SP1044 supplied from Dansk Voksfabrik) as spraying agent.

The spraying agent was supplied to the spraying tower with a hot air

stream of 85°C and in an amount of 3-4 kg/min.

In order to ensure a rapid evaporation of water and to expedite the drying of the surface of the fine drops formed by spraying, additional air having a temperature of 85°C was supplied to the spraying tower in the spraying zone.

The spraying of the emulsion was effected by means of a disc sprayer having 2300 rotations/min. The temperature in the spraying tower was 55°C.

The emulsion drops having freshly solidified surfaces were transferred together with the Hartwachs SP1044 spraying agent to a fluidized bed and maintained fluidized for about 3 hours by the introduction of drying air having a temperature of 63°C. During the drying the temperature of the encapsulated product did not exceed 40°C and the excess fine Hartwachs SP1044 particles were recycled to a hopper.

The ready-dried product was then removed and sieved into two fractions, viz. a product fraction of 125-450 µm and a coarse fraction.

The product obtained was a free-flowing product having a high particle density.

Example 7

Evaluation of encapsulation of flavourants

A taste panel consisting of 10 persons tasted 6 different pieces of chewing gum and reported the time within which the flavourant could be tasted after the point of start. Table 1 below shows the average values in seconds obtained for three different flavour products in chewing gum formulations comprising the flavour products in encapsulated and non-encapsulated form, respectively. In addition to 1-2% flavourant the chewing gum formulation further comprises about 20% sugar, about 24% gum base and about 56% paraffin.

Preparation of chewing gum containing flavour oil or coated flavour

24 g of Gum base was melted and 56 g of melted paraffin and 20 g of sugar were added under vigorous stirring. 1.4-10 g of Flavourant was added under stirring at 48-50°C. The flavourant was used in an amount corresponding to 1.4 g pure oil. In the addition of microencapsulated flavourant the amount added corresponds to about 1-2% by weight of the finished chewing gum.

Stirring was continued until solidification occured. The chewing gum which had started to solidify was poured out onto a glass plate and was pressed flat by another glass plate. The chewing gum was then allowed to stand for the night. The next day the chewing gum was cut into small pieces of 0.95-1.10 g.

Table 1

| Flavourant | Initial taste in sec. after beginning of tasting | |
|---|---|---|
| Mintflavour | 5 | Not encapsulated |
| Encapsulated mintflavour | 90 | Encapsulated according to example 4 |
| Spearmint flavour | 2 | Not encapsulated |
| Encapsulated spearmint flavour | 25 | Encapsulated according to example 5 |
| Orange oil | 1 | Not encpasulated |
| Encapsulated orange oil | 23 | Encapsulated according to example 6 |

The results in table 1 show that by using flavourants which are encapsulated according to the invention in chewing gum a considerable extension of flavour start is obtained compared to chewing gum which contains non-encapsulated flavourants.

Example 8

Microencapsulation of extract of parsley oil

600 g of Water was heated to 35°C, 330 g of fish gelatine and 330 g of saccharose were added and dissolved under stirring.

50 g of Extract of parsley oil was emulsified into the fish gelatine/saccharose mixture. The comminution of the oil resulted in an average oil drop size of 0.7 µm.

The viscosity was adjusted to 112 cP at 35°C by dilution with water.

The emulsion was introduced into a spraying tower at a velocity of 1.5 l/min. and sprayed therein in the form of fine drops in a cloud of fine particles of corn starch which was supplied simultaneously time to the tower from a hopper. The spraying agent was introduced into the spraying tower at a velocity of 1:5 kg/min. The spraying was effected by a disc sprayer having 1200 rotations/min. The temperature in the spraying tower was 68°C and during the passage down through the tower the surface of the fine drops formed by spraying solidified.

The emulsion drops having freshly solidified surfaces were transferred to a fluidized bed and maintained fluidized for about 8 hours at a temperature of 30°C. During the drying the excess corn starch was recycled to the hopper.

The ready-dried product was removed and sieved into two fractions, viz. a product fraction of 100-500 µm and a coarse fraction of more than 500 µm.

A water vapour distillation according to the Clevenger Method showed a parsley oil content of 5.2%.

Example 9

Microencapsulation of extract of lovage oil

600 g of Water was heated to 40°C, 330 g of fish gelatine and 330 g of saccharose were added and dissolved under stirring.

50 g of Extract of lovage oil was emulsified into the fish gelatine/saccharose mixture. The viscosity was adjusted to 116 cP at 40°C by dilution with water.

The emulsion was introduced into a spraying tower at a velocity of 1.5 l/min. and sprayed therein in the form of fine drops in a cloud of fine particles of corn starch which was supplied simultaneously to the tower from a hopper. The spraying agent was introduced into the spraying tower at a velocity of 1.5 kg/min. The spraying was effected by a disc sprayer having 1200 rotations/min.

The temperature in the spraying tower was 65°C and during the passage down through the tower the surface of the fine drops formed by spraying solidified.

The emulsion drops having freshly solidified surfaces were transferred to a fluidized bed and maintained fluidized for about 8 hours a temperature of 30°C. During the drying the excess corn starch was recycled to the hopper. The ready-dried product was removed and sieved into two fractions, viz. a product fraction of 100-500 µm and a coarse fraction of more than 500 µm.

A water vapour distillation according to the Clevenger Method showed a lovage oil content of 6.3%.

Example 10

Microencapsulation of extract of dill oil

3000 g of Water was heated to 65°C, 1994 g of 0-10 Bloom gelatine and 1994 g of saccharose were added and dissolved under stirring. The solution was deaerated under vacuum at 0.81 bar for 2 hours at

65°C. The vacuum in the container was decreased by introduction of nitrogen and 900 g of extract of dill oil added with 1.28 g of mixtocopherol was emulsified into the gelatine/saccharose mixture to an oil drop size of 1 µm.

The viscosity was then adjusted to 80 cP at 62°C by the addition of water which was deaerated for oxygen.

The emulsion was introduced into a spraying tower at a velocity of 1.5 l/min. and sprayed therein in the form of fine drops (about 1.1-0.3 mm) in a cloud of fine particles of spraying agent in the form of corn starch which was supplied simultaneously to the tower at a velocity of 2 kg/min. The spraying was effected by a disc sprayer having 2000 rotations/min.

The temperature in the spraying tower was 65°C and during the passage down through the tower the surfaces of the emulsion drops solidified.

The emulsion drops having freshly solidified surfaces were transferred to a fluidized bed and maintained fluidized for about 3 hours at a temperature of 40°C.

During the drying the excess corn starch was recycled to the hopper.

The ready-dried product was then removed and sieved into two fractions, viz. a product fraction of 100-500 µm and a coarse fraction of more than 500 µm.

A water vapour distillation according to the Clevenger Method showed a content of extract of dill oil of 14.7%. An analysis by means of HPLC (high performance liquid chromatography) showed a content of extract of dill oil of 15.1%.

Example 11

750 g of Water was heated to 65°C, 312 g of 240 Bloom type A gelatine and 115 g of glycyrrhizin having a concentration of 90.7% glycyrrhizin were added and dissolved under stirring.

The viscosity was adjusted to 160 cP at 65°C by dilution with water.

The solution was introduced into the spraying tower at a velocity of 1.5 l/min. and sprayed therein in the form of fine drops in a cloud of fine carnauba wax particles (carpol 1305) which were supplied at the same time to the tower from a hopper.

The spraying agent was introduced into the tower at a velocity of about 2 kg/min. The spraying was effected by a disc sprayer having a velocity of 1200 rotations/min and the temperature in the spraying tower was 65°C.

The emulsion drops having freshly solidified surfaces were transferred together with the carnauba wax spraying agent to a fluidized bed and maintained fluidized for about 3 hours by the introduction of a drying air having a temperature of 38°C.

The excess fine carnauba wax powder was recycled to the hopper. The ready-dried product was then removed and sieved into two fractions, viz. a coarse fraction and a product fraction of 125-420 μm.

The product can be used as a flavourant in confectionery.

Patent Claims

1. A process for preparing microcapsules containing a flavourant embedded in a matrix material comprising spraying an aqueous emulsion, suspension or solution of the flavourant and the matrix material under the supply of air to form a free-flowing product, characterized in that the spraying is effected under the supply of air having a temperature of 50 to 120°C while simultaneously introducing a spraying agent, and in that the sprayed product is subjected to a further drying in a fluidized bed at a temperature between 0 and 120°C.

2. A process according to claim 1, characterized in that an emulsion of an oil containing a flavourant and aqueous colloid solution is sprayed under the supply of air having a temperature of from 60° to 85°C, and that the sprayed product is subjected to an additional treatment in a fluidized bed at a temperature between 20 and 85°C.

3. A process according to claims 1 or 2, characterized in spraying an emulsion comprising oil drops containing a flavourant having a diameter of from 0.2-10 μm.

4. A process according to any of the preceding claims, characterized in using a colloid, such as gum arabic, modified starch, glucose sirup, dextrines and gelatine or mixtures thereof as matrix material.

5. A process according to claim 4, characterized in using gelatine having a Bloom strength of from 0-300, preferably from 30-300, as matrix material.

6. A process according to any of the preceding claims, characterized in that the matrix material further comprises wax.

7. A process according to claim 6, characterized in that the wax is carnauba wax, candelila wax, paraffin wax, a wax marketed under the name Hartwachs SP1044 and synthetic waxes.

8. A process according to any of the preceding claims, c h a r a c t e r i z e d in using as spraying agent modified starch, casein, tricalcium phosphate, lactose, ethyl cellulose, coagulated albumin, stearic acid, cured gelatine, calciumstearat and sodiumstearat, hydrogenated castor oil, polyoxide, talc, vegetable wax and paraffin wax or mixtures thereof in a proportion of at least 1 part by weight of spraying agent per part by weight of emulsion, suspension or solution.

9. A process according to any of the preceding claims, c h a r a c t e r i z e d in using a proportion of from 1/99 to 2/3 parts by weight of flavourant per part by weight of matrix material.

10. A process according to any of the preceding claims, c h a r a c t e r i z e d in using from 1-15% by weight of flavourant, from 20-35% by weight of colloid and from 1-8% by weight of wax in the emulsion or suspension.

11. Microcapsules comprising a flavourant embedded in a matrix material, c h a r a c t e r i z e d in that the microcapsules are prepared by spraying an aqueous emulsion, suspension or solution of flavourants and the matrix material under the supply of air having a temperature of from 50 to 120°C while simultaneously introducing a spraying agent, and that the sprayed product is subjected to an additional drying in a fluidized bed at a temperature between 0 and 120°C.

12. Microcapsules prepared according to claims 6, 7 or 8, c h a r a c t e r i z e d in that wax constitutes from 5-20% by weight of the microcapsule.

13. Microcapsules according to claims 11 or 12, c h a r a c t e r i z e d in that the flavourant constitutes from 0.1 - 40% by weight of the finished microcapsules.

14. Microcapsules according to claims 11, 12 or 13, c h a r a c t e r i z e d in that the finished microcapsule has a particle size of from 50 to 1000 μm.

15. Microcapsules according to claims 11, 12, 13 or 14, c h a r a c t e r i z e d in that the particle density varies within the range of 1.0-1.5 g/ml.

16. A chewing gum containing microcapsules comprising a flavourant embedded in a matrix material, c h a r a c t e r i z e d in that it contains microcapsules according to claim 11.

17. A chewing gum containing microcapsules comprising a flavourant embedded in a matrix material, c h a r a c t e r i z e d in that it contains microcapsules according to claim 12.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/DK 91/00133

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: B 01 J 13/04, A 23 G 3/30

## II. FIELDS SEARCHED

Minimum Documentation Searched[7]

| Classification System | Classification Symbols |
|---|---|
| IPC5 | B 01 J; A 23 G |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

## III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

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| X | EP, A1, 0070719 (UNILEVER PLC ET AL) 26 January 1983, see page 5, line 27 - page 7, line 29; page 9, line 5 - page 10, line 2 -- | 1-5,8-11,13-15 |
| A | US, A, 4695463 (YANG ET AL) 22 September 1987, see column 5, line 32 - line 40; column 6, line 38 - column 8, line 17; column 11, line 35 - column 12, line 37 -- | 1-17 |
| X | US, A, 4519961 (SCHUMACHER ET AL) 28 May 1985, see column 2, line 42 - line 49; column 4, line 26 - line 49; claim 6 -- | 1-5,8-11,13-15 |

* Special categories of cited documents:[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

## IV. CERTIFICATION

| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report |
|---|---|
| 22nd August 1991 | 1991 -08- 27 |
| **International Searching Authority** | **Signature of Authorized Officer** |
| SWEDISH PATENT OFFICE | Eva Iversen Hasselrot |

Form PCT/ISA/210 (second sheet) (January 1985)

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

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# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/DK 91/00133

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the Swedish Patent Office EDP file on 91-06-27
The Swedish Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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